

**A PROCESS FOR REMOVAL OF ORGANIC SULPHUR FROM HIGH
SULPHUR COAL AND A DEVICE THEREFOR**

5 The present invention relates to a process for removal of organic sulphur from high sulphur coal and a device therefor.

The invention finds its usage in utilizing for power generation in thermal coal plants, briquette making, active carbon and reduction of pollution.

10

Sulphur included within the coals, make them undesirable for use without expensive and efficient reducing equipment to recover the products of sulphur by combustion. One of the products of combustion is sulphur dioxide, which is considered to be a highly undesirable pollutant of the atmosphere. It is generally recognized that sulphur in coal is present in various forms. There are three basic forms, namely; pyritic sulphur, sulphate sulphur and organic sulphur. The pyritic sulphur is sulphur combined with iron. The sulphate sulphur is generally of a minute quantity, that is, it usually constitutes 1% or 2% of all the sulphur in a given coal specimen. Organic sulphur is sulphur, which is combined, in an organic compound with the carbon of the coal. Organic sulphur is generally found to be difficult to remove from coal. In US Patent No.4233034 the problem which confronts most investigators is how to remove organic sulphur from coal. The patent discloses a process for removing sulphur from coal. But process does not distinguish between the removal of organic sulphur from the removal of pyritic sulphur, but the teaching is simply directed to a process of removing sulphur without distinction of what type of sulphur is removed. It is known to remove pyritic sulphur efficiently from coal. The problem revolves around removing the organic sulphur efficiently.

15
20
25

Reference may be made to US Patent No: 4441886 wherein an improved process for removing organic sulphur from coal has been described. The process includes a plurality of steps. Coal containing organic sulphur is broken down into granules. The granules of coal may, but need not necessarily be treated to remove substantially all of the water

30

contained in the coal so that the coal is dry. The dried coal is then mixed with a selected quantity of ethyl alcohol which contains less than 4% water. The quantity of ethyl alcohol is such in each instance that the coal ethyl alcohol mixture contains less than 61.7% solids by weight. The mixture of coal and ethyl alcohol is placed in a reaction vessel, and
5 temperature and pressure in the vessel are raised above the critical temperature and pressure of ethyl alcohol. The mixture is maintained at a temperature and pressure above the critical temperature and pressure for a period of time. Any gas generated is collected and the coal solids of the mixture are separated from the liquid. Prior art search for removal of organic sulphur from coal and a device therefor was made based on literature
10 survey and patent databases, which did not yield any relevant references

The main object of the invention is to provide a device for removal of organic sulphur from high sulphur coal which obviates the drawbacks as detailed above.

15 Another object of the invention is a process for removal of organic sulphur from high sulphur coal.

Accordingly the present invention relates to a device for reduction of organic Sulphur from high Sulphur coal which comprises a reactor essentially consisting of three heating
20 zones such as steam heating zone capable of maintaining a temperature in the range of 450-500 degree centigrade, a promoter zone capable of maintaining a temperature of the order of 950-1100 degree centigrade and reaction zone capable of maintaining a temperature in the range of 900-950 degree centigrade, the said reactor being placed inside a tabular furnace capable of providing the above said temperature zones in the said
25 reactor, the said furnace with reactor inside being enclosed in a movable cabinet, the said reactor and furnace being provided with energy regulator and indicators.

In an embodiment of the present invention relates to a device wherein the tabular furnace is made up of Silliminite and insulated by quartz wool.

30

In an another embodiment of the present invention relates to a process for removal of organic sulphur from high sulphur coal using the device as claimed in claim1 which comprises of heating the promoter zone (containing the promoter) at a temperature in the range of 1100±50 degree Celsius and steam zone at a temperature in the range of 450 to 500 degree Celsius, crushing the input coal to -72 mesh BS and feeding into the reaction zone, producing steam in a flask, preferably made of glass and passing through the reactor, maintaining the temperature at 900 degree Celsius for about 1 hour, after it attains a temperature of 900 degree Celsius, passing the gas evolved from the reactor through a series of bubblers, preferably made of glass, containing ammoniacal cadmium chloride solution, cooling the furnace to room temperature and discharging the product coke/char.

In another embodiment of the invention relates to a process as claimed in 2 wherein the promoter used is mixture of copper-iron turnings in the ratio of 1:9.

15

In yet another embodiment of the present invention relates to a process as claimed in 2 & 3 wherein the rate of rise in temperature in promoter zone and reaction zone is 5 degree Celsius per minute.

20 In the process the promoter zone is heated initially at a temperature in the range of 950 to 1000 degree Celsius at the rate of 5 degree Celsius rise in temperature and steam zone at a temperature in the range of 450 to 500 degree Celsius at the rate of 5 degree Celsius rise in temperature. The input coal is crushed -72 mesh BS and fed into the reaction zone. Steam is produced in a round bottom flask and passed through the reactor. When temperature attains 900 degree Celsius, it is maintained at that temperature for a time period in the range of 0.5 to 2 hours. The gas evolved is passed through a series of glass bubblers containing ammoniacal cadmium chloride solution resulting in formation of yellow precipitate of cadmium sulphide inside the bubblers. The furnace is cooled to room temperature and the product coke/char is discharged.

30

In the drawing(s) accompanying this specification, the different components of the device are:

1. Silica Reactor
2. Steam Heating Zone
- 5 3. Promoter Zone
4. Reaction Zone
5. Insulation with Quartz Wool
6. Furnace, made of silliminite
7. Insulation with Quartz Wool
- 10 8. Frame for insertion of silliminite
furnace with silica reactor inside.

The present invention provides a process for a device for removal of organic sulphur from high sulphur coal which comprises of a tubular furnace (6) , made of silliminite and
15 insulated by quartz wool (5 & 7), having outer diameter of 250 mm, internal diameter of 40 mm, length 650 mm with tube bore of 37.5 mm; a reactor, preferably made of silica (1), having three heating zones viz. i) steam heating zone (2) of 17mm long, for maintaining a temperature in the range of 450 to 500 degree Celsius, ii) Promoter heating zone (3) of 250 mm length, for maintaining temperature in the range of 950 to 1200
20 degree Celsius, iii) reaction zone (4) of 200 mm length, for temperature in the range of 300 to 1100 degree Celsius; the said furnace is fitted with two chrome-aluminium thermocouples, digital temperature indicators with multi point switch and three energy regulators (9) to control the temperatures of the three zones; the said furnace (6) is fitted with rollers to move freely on rail tracks fitted in a rectangular control cabinet, inside
25 which energy regulators and indicators are fitted; a reactor(1), preferably made of silica, of length 940 mm, internal diameter of 21 mm and outer diameter of 23 mm is inserted into the said furnace; a frame (8), preferably made of mild stainless steel having length of 750 mm, width of 325 mm and depth of 325 mm, for accommodating the said furnace with the silica reactor(1) inside.

30

The present invention provides a process for removal of organic sulphur from high sulphur coal which comprises of heating the promoter zone at a temperature in the range of 300 to 1100 degree Celsius and steam zone at a temperature in the range of 450 to 500 degree Celsius; crushing the input coal to -72 mesh BS and feeding into the reaction zone; producing steam in a flask, preferably made of glass and passing through the reactor; maintaining the at the temperature 900 Celsius for a time period in the range of 0.5 to 2 hours, passing steam after it attains a temperature of 900 degree Celsius; passing the gas evolved from the reactor through a series of bubblers, preferably made of glass, containing ammoniacal cadmium chloride solution; cooling the furnace to room temperature and discharging the product coke/char.

In an embodiment of the present invention the promoter used is mixture of copper-iron turnings in the ratio of 1:9.

In another embodiment of the present invention the rate of rise in temperature in promoter zone and reaction zone is 5 degree Celsius per minute.

Novelty of this invention lies in removing of organic sulphur from high sulphur coal, which is otherwise unutilised worldwide Organic sulphur is sulphur, which is combined, in an organic compound with the carbon of the coal. Organic sulphur is generally found to be difficult to remove from coal. Sulphur dioxide, which is considered to be a highly undesirable pollutant of the atmosphere. Present invention removes 79.77 % of organic sulphur from coal, which is a break through.

The following examples are given by way of illustration of the present invention and should not be construed to limit the scope of the present invention.

Example-1

The Promoter zone and steam zone was heated initially up to 1100 degree Celsius & 450 degree Celsius respectively with increase in temperature being 5 degree Celsius per minute. 10 grams of coal was crushed to -72 mesh BS and fed into the reaction zone. 9 g

of iron and 1 g of copper turnings were taken in the promoter chamber. Steam was produced in a round bottom flask and passed through the reactor. When the furnace temperature attained 900 degree Celsius, it was maintained at that temperature for one hour. The gas evolved was passed through a series of glass bubblers containing ammoniacal cadmium chloride solution, which resulted in formation of yellow precipitate of cadmium sulphide inside the bubblers. The furnace was cooled to room temperature and the product coke/char was discharged. While the original coal contained 5.39% of organic Sulphur, after the above process it was reduced to 1.17%, resulting overall reduction in organic Sulphur as 78.29 %.

10

Example-2

The promoter zone and steam zone was heated initially up to 1000 degree Celsius & 400 degree Celsius respectively with increase in temperature being 5 degree Celsius. 10 g of coal was crushed to -72 mesh BS and fed into the reaction zone. 9 g of iron and 1 g of copper turnings were taken in the promoter chamber. Steam was produced in a round bottom flask and passed through the reactor. When the furnace temperature attained 900 degree Celsius, it was maintained at that temperature for one hour. The gas evolved was passed through a series of glass bubblers containing ammoniacal cadmium chloride solution, which resulted in formation of yellow precipitate of cadmium sulphide inside the bubblers. The furnace was cooled to room temperature and the product coke/char was discharged. While the original coal contained 5.39% of organic Sulphur, after the above process it was reduced to 0.88 % resulting overall reduction in organic Sulphur as 79.77%.

25

Example-3

The promoter zone and steam zone was heated initially up to 900 degree Celsius & 550 degree Celsius respectively with increase in temperature being 5 degree Celsius. 10 g of coal was crushed to -72 mesh BS and fed into the reaction zone. 9 g of iron and 1 g of copper turnings were taken in the catalyst chamber. Steam was produced in a round bottom flask and passed through the reactor. When the furnace temperature attained 900

30

degree Celsius, it was maintained at that temperature for one hour. The gas evolved was passed through a series of glass bubblers containing ammoniacal cadmium chloride solution, which resulted in formation of yellow precipitate of cadmium sulphide inside the bubblers. The furnace was cooled to room temperature and the product coke/char was discharged. While the original coal contained 5.39% of organic Sulphur, after the above process it was reduced to 1.51% resulting overall reduction in organic Sulphur as 71.98 %.

Example-4

The promoter zone and steam zone was heated initially up to 600 degree Celsius & 400 degree Celsius respectively with increase in temperature being 5 degree Celsius. 10 grams of coal was crushed to -72 mesh BS and fed into the reaction zone. 9 g of iron and 1 g of copper turnings were taken in the catalyst chamber. Steam was produced in a round bottom flask and passed through the reactor. When the furnace temperature attained 900 degree Celsius, it was maintained at that temperature for one hour. The gas evolved was passed through a series of glass bubblers containing ammoniacal cadmium chloride solution, which resulted in formation of yellow precipitate of cadmium sulphide inside the bubblers. The furnace was cooled to room temperature and the product coke/char was discharged. While the original coal contained 3.24% of organic Sulphur, after the above process it was reduced to 2.32 %, resulting overall reduction in organic Sulphur as 28.39 %.

The main advantages of the present invention are:

1. The process is very simple and eco-friendly.
2. The process involves most commonly used materials and hence is cost effective.